

PROCESSING FEEDS CONTAINING HEMICELLULOSE EXTRACT

by

Montford Lee Carpenter

B.S., Kansas State University, 1965

---

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

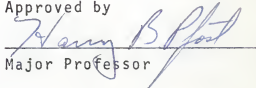
Department of Grain Science and Industry

KANSAS STATE UNIVERSITY

Manhattan, Kansas

1968

Approved by



Major Professor

LD  
4668  
74  
1948  
C392  
12

## TABLE OF CONTENTS

	PAGE
INTRODUCTION. . . . .	1
REVIEW OF LITERATURE. . . . .	3
Physical Properties . . . . .	3
Mixing. . . . .	4
Pelleting . . . . .	7
METHODS AND MATERIALS . . . . .	8
Mixing Studies. . . . .	8
Preliminary studies . . . . .	8
Development of the chloride tracer method . . . . .	10
Experimental design . . . . .	14
Preparation of liquid ingredients used in mixing studies. . . . .	15
Equipment and procedures. . . . .	16
Pelleting Studies . . . . .	28
Equipment and procedures. . . . .	28
Effect of high levels of hemicellulose extract. . . . .	31
Effect of low levels of hemicellulose extract . . . . .	33
Comparison of dried hemicellulose extract and lignin binder. . . . .	33
RESULTS AND DISCUSSION. . . . .	37
Mixing Studies. . . . .	37
Pelleting Studies . . . . .	46
Effect of high levels of hemicellulose extract. . . . .	46
Effect of low levels of hemicellulose extract . . . . .	49

Comparison of dried hemicellulose extract and a lignin binder. . . . .	49
CONCLUSIONS. . . . .	52
Mixing Studies . . . . .	52
Pelleting Studies. . . . .	53
LITERATURE CITED . . . . .	54
ACKNOWLEDGEMENTS . . . . .	55

## INTRODUCTION

Hemicellulose extract is the concentrated soluble material obtained from the steam treatment of wood at elevated temperatures and pressures, without the use of acids, alkalies, and salts. It is a by-product of an industrial process for the production of hardboard. It is produced in liquid form and sold under the trade name of Masonex<sup>R</sup>. A spray dried product is also produced and sold under the trade name of Dried Masonex<sup>R</sup>. The liquid product is manufactured at plant locations in Laurel, Mississippi, and Ukiah, California. The dried product is produced in the Laurel, Mississippi plant. Both products are produced by Masonite Corporation.

Liquid and dried hemicellulose extract are used as an energy source in livestock feeds in much the same way as molasses.

The purpose of this research was to study the properties of feed mixtures containing hemicellulose extract during mixing and pelleting. Since liquid hemicellulose extract is used in livestock feeds in a manner similar to cane molasses, a comparison was made between the mixing properties of these two liquids with a dry feed ingredient. Much of this research was devoted to the development of procedures for evaluating the mixing of liquid and dry ingredients.

Feed rations containing hemicellulose extract and cane molasses were pelleted to compare pellet durability and power

requirements. Pelleting studies were also conducted to compare dried hemicellulose extract with another pellet binding agent.

## REVIEW OF LITERATURE

### PHYSICAL PROPERTIES

Nordstedt (5) reported on the physical properties of liquid hemicellulose extract from Mississippi and California. The viscosity of hemicellulose extract from Mississippi is somewhat higher than the viscosity of cane molasses. The viscosity of hemicellulose extract from California is similar to that of cane molasses. Nordstedt determined the viscosity of hemicellulose extract at different temperatures and upon dilution with water and compared these characteristics with those of cane molasses. The effects of enzymes and surfactants on viscosity were also investigated.

Cross (1) reported on the manufacture of hemicellulose extract. Wood chips are charged into a vertical digester, cooked by direct high pressure steam, then suddenly released and blown to a collector cyclone. The coarse fiber in a water slurry is further refined with disk grinders. The milled pulp is washed in a triple-stage countercurrent vacuum-drum washer and further processed into hardboard. The wash water from the first stage contains four to five per cent dissolved and colloidal solids. This wash liquor is concentrated in spray driers to about 45 per cent solids. A vertical tube, falling-film evaporator concentrates the material from the spray drier to 65 per cent solids. The material from the evaporator is normally neutralized to a pH of 5.7 to 5.9. The 45-per cent-solids material is dried

in a spray drier to form the dried hemicellulose extract.

### MIXING

An ideal feed mixture should have all ingredients uniformly distributed throughout the mass of material so the animal receives a uniform amount of each nutrient each day. As a liquid is being mixed with solid feed ingredients, dispersion of the liquid is accomplished in two ways. First, part of the liquid is absorbed by the solid particles. Heideman (2) gives three factors that affect absorption; these are: temperature of the liquid and other ingredients, moisture content of the solid ingredients, and the time of contact. The second means of dispersion of a liquid in a feed mixture is referred to in the literature as "balling." Lucas (3) states that ideal mixing is done when the liquid is blended to a point where it is divided into very small dust covered balls not visible under ordinary inspection. Ordinarily, liquid mixing is a combination of absorbing and balling. In the process, the appearance of large dust covered balls of liquid indicates improper mixing. Heideman (2) states that most mixers will produce feeds with less than one per cent of these balls being formed. There is no suggestion in the literature as to ways of determining the size of dust covered balls that may be in a feed mixture.

Stroup (8) reviews the advantages and disadvantages of several types of mixers that are used for blending liquids into solid feed material. The double agitator continuous mixer is

used where high liquid percentages and high capacities are desired. The single agitator continuous type is used also for high capacities but lower liquid levels. Both of these types are usually designed for easy cleanout of molasses buildup. Horizontal and vertical batch mixers are usually used for low liquid percentages; cleanout is a problem in both of these mixers.

For good dispersion of liquid in a feed mixture, Heideman (2) recommends the following methods for adding liquids to a mixer. The first method is to discharge the liquid line onto a perforated plate at a point where the solid material enters the mixer. The liquid can then drop through the perforations onto the feed. The next method is to force the liquid into the mixer by means of nozzles or holes in the mixer shaft. Another method is to spray the liquid through nozzles in a ring around the point where the solid ingredients enter the mixer. The fourth method is to drop the liquid on a horizontal disk revolving at a high speed; the liquid is then thrown off of the disk and onto the feed which is falling in a cylindrical stream around the disc.

Because of the nature of the liquids (foreign matter in them) and the tendency of dust to accumulate, these methods require considerable maintenance and cleaning. Also because of wide variations in the quantity of liquids being added, the effectiveness of these methods on low rates, and high pressure buildup on high rates, makes their advantages questionable. For these reasons Stroup (8) has reported that most mills are using



open pipes for liquid introduction into a mixer. While dispersion is not as good with this method, it does not require the attention and maintenance that the other methods require.

The usual procedure for determining the distribution of ingredients in a feed mixture is first to collect a series of samples of the mixed feed. These samples are then assayed for certain properties. Pierce (7) reported on assay methods commonly used. He classified them into five categories: chemical assays, mineral assays, and and specific nutrient assays such as vitamin assays, drug assays and tracer methods.

After the assay values are obtained, most authors use the statistical measure of coefficient of variation to place an index on the uniformity of a feed mixture. Coefficient of variation is defined as

$$V = \frac{s}{m} \times 100$$

where V = per cent coefficient of variation

s = standard deviation of assay values

m = mean of assay values.

The standard deviation is determined from the following formula:

$$s = \sqrt{\frac{\sum x_i^2 - \frac{1}{n} (\sum x_i)^2}{n-1}}$$

where  $x_i$  = assay value of the i'th sample

n = number of samples assayed.

Of course, the mean is determined from

$$m = \frac{\sum_{i=1}^n x_i}{n}$$

Pfost (6) reports that the number of samples must be large enough to minimize errors from too few samples. He selected a minimum of ten samples as being appropriate for testing feed mixtures. Pfost also describes the effect of the Poisson distribution due to a limited number of particles in a sample. To keep the error due to this effect below 3% at least 900 particles of the material to be assayed must be included in the sample. Thus the interpretation of assay results must consider sample size and number of particles expected in the sample.

Pfost (6) concludes that with good mixing the total coefficient of variation should not exceed 20 per cent. If adequate numbers of particles are present in the samples, mixer tolerance can be reduced. Usually with most ingredients, the coefficient of variation that may be allowed due to poor mixing could range as high as 5 to 20 per cent.

#### PELLETING

Nordstedt (5) pelleted a beef cattle ration containing 10% of either liquid hemicellulose extract or cane molasses. He found no significant difference in the pellet durability index (9) and power required for pelleting these two rations.

## METHODS AND MATERIALS

### MIXING STUDIES

The purpose of these studies was to evaluate the mixing characteristics of hemicellulose extract with dry feed ingredients. Because the viscosity of hemicellulose extract from Mississippi is greater than that of cane molasses and because hemicellulose extract is used in livestock feed in the same way as cane molasses, properties of these two liquids were of primary interest. Since hemicellulose extract from California has a viscosity similar to or less than that of cane molasses, it was not studied. The mixing properties of dried hemicellulose extract were not studied. Before a comparison could be made between the mixing properties of hemicellulose extract from Mississippi and cane molasses, a procedure was needed that would evaluate the quality of a mixture of liquid with dry feed ingredients. Preliminary studies were carried out using two simple techniques for evaluating uniformity.

Preliminary Studies. The first technique was that of using moisture as a tracer. Since cane molasses contains 20% to 30% moisture and liquid hemicellulose extract contains 35% moisture, it was reasoned that the distribution of moisture in a feed mixture could serve as an indication of the distribution of hemicellulose extract or cane molasses in the feed mixture. If this were the case, it would be a simple

procedure to dry a series of samples from a feed mixture and thus have an evaluation of the distribution of the liquid ingredient in the feed.

The mention of "balling" in the literature (2,3) suggested another procedure that might be used to evaluate mixing of liquid ingredients. The amount of these dust covered liquid balls or lumps in a feed mixture could be determined by a sieving process. The amount of balls would then possibly be an indication of the uniformity of the mixture.

Preliminary studies using the moisture tracer method indicated that even though the method might serve for a study of feed materials with a high level of molasses, say 20% to 30%, it was not an accurate indication of degree of mixing for feed material with less than 20% molasses being added. This method was further restricted by the fact that it could not be used after the mixture was allowed to dry. Preliminary studies using a sieving analysis to determine the amount of liquid balls did not show that reproducible results could be obtained. The results also did not correspond to those obtained using the moisture tracer method. It became evident that a more reliable method was needed to determine the distribution of liquid ingredients such as hemicellulose extract and cane molasses in a feed mixture.

Development of the Chloride Tracer Method. A common method for evaluating mixer performance is that of using ordinary sodium chloride as the tracer. Luhman's (4) rapid method for potentiometric detection of soluble chlorides in feed materials is a quantitative test which can be used to determine the amount of salt in individual samples. This method has been used on feed mixtures consisting of dry ingredients. The possibility of extending this method for use in the determination of the distribution of liquid ingredients in a feed mixture was evaluated.

If a chloride compound could be dissolved in hemicellulose extract and cane molasses, it could be used as a tracer to evaluate the mixing characteristics of these liquids. The use of this method had one important restriction: the chlorine compound that was dissolved in the liquid could not change the physical properties of the liquid because, if it did, the mixing characteristics of that liquid would be altered. The first step in developing this method was that of finding a chloride compound that would not affect the physical properties of hemicellulose extract and cane molasses. The physical properties considered were viscosity and surface tension. It was assumed that if a chloride compound could be dissolved in hemicellulose extract and cane molasses without changing their viscosity or surface tension, the mixing properties of these two liquids would remain unchanged.

A Brookfield Model LVT "Synchro-Lectric"<sup>R</sup> spindle

viscometer was used to determine the viscosities of the liquids. The surface tensions were determined with a submerged ring type tensiometer.

Even though Luhman's method is usually used for common livestock salt, or sodium chloride, it can be used for other chloride compounds as well. Experiments with different amounts and combinations of ammonium chloride, potassium chloride, and sodium chloride dissolved in hemicellulose extract and cane molasses showed that these compounds would give the desired results. Table 1 shows the results of these experiments. Different levels of potassium chloride dissolved in hemicellulose extract caused no significant change in either its viscosity or surface tension. Different amounts of a mixture of 50% ammonium chloride and 50% sodium chloride caused no change in the viscosity and surface tension when dissolved in cane molasses.

In the following, the use of the term chlorides refers to potassium chloride in the case of hemicellulose extract and a mixture of equal amounts of ammonium chloride and sodium chloride in the case of cane molasses.

Using the chlorides indicated dissolved in the liquids as tracers, it was now possible to determine the distribution of hemicellulose extract and cane molasses in a feed mixture. It was assumed that if the chloride was uniformly distributed in the liquid ingredients, the distribution of these liquid ingredients in a feed mixture would be the same as the distribution of the chloride in the feed mixture as determined by

Table 1. Effect of Chloride Compounds on the Viscosity and Surface Tension of Hemicellulose Extract and Cane Molasses.

Treatment	Level (% by wt.)	Viscosity (cps. at 29 C)	Surface Tension (dynes/cm.)
Hemicellulose Extract			
control	--	3050	56.0
NaCl	1.67	3040	
	2.5	3980	
	5.0	5170	
	10.0	7850	
KCl	1.67	3030	55.4
	2.0	3020	55.3
	2.5	2860	55.4
	3.3	2770	55.9
	5.0	3020	56.0
	10.0	3270	56.1
NH <sub>4</sub> Cl	10.0	2190	
Cane Molasses			
control	--	716	58.2
NaCl	1.67	836	
	2.5	1080	
	5.0*	1140	
	10.0*	1420	
NH <sub>4</sub> Cl & NaCl (equal parts)	1.67	680	57.7
	2.0	720	57.0
	2.5	716	57.8
	3.3	700	56.7
	5.0	702	56.5
KCl	10.0*	700	
NH <sub>4</sub> Cl	10.0	388	

\*In these treatments the chloride compounds were not completely dissolved in the liquid.

Luhman's method for potentiometric detection of soluble chlorides in feed materials.

Samples of known amounts of the chloride compounds were dissolved in hemicellulose extract and cane molasses. These samples were analyzed for chloride using the potentiometric method. Coefficients of variation of the chloride contents of 10 samples analyzed from each liquid were 0.67% for hemicellulose extract and 0.54% for cane molasses. These coefficients of variation were low enough to indicate a uniform distribution of the chloride in both liquids. The chloride analysis of the chlorides dissolved in cane molasses always showed a larger amount of chloride present than the amount that had been added. This indicated that cane molasses alone contained some chloride. An analysis of cane molasses showed that it contained about 2.25% chloride expressed as per cent chloride compound (ammonium chloride and sodium chloride). Hemicellulose extract on the other hand contained essentially no chloride.

Luhman reported accurate results on feeds with a salt content as low as 0.75%. To have at least this amount of chloride compound present in the total feed mixture, it was necessary to have 7.5% chloride compound dissolved in the liquid ingredient when this liquid was at a 10% level in the total feed mixture. By dissolving 7.5% potassium chloride in hemicellulose extract and 5.4% of the ammonium chloride and sodium chloride mixture in cane molasses, the necessary chloride level was obtained in both liquids. The lower amount of



chloride compound dissolved in cane molasses was permitted because of the chloride already present in the molasses.

Known levels of these liquid solutions were mixed with a dry ingredient (wheat bran) in the laboratory and analyzed. The chloride values obtained had a coefficient of variation of less than 2% which indicated the assay error was sufficiently small so that the test would be valid when used in actual mixing studies. There was a small amount of soluble chloride (0.10%) detected in the wheat bran but this amount had little affect on the final results.

Experimental Design. As mentioned previously, the primary purpose of this research was to compare the mixing characteristics of hemicellulose extract and cane molasses. Also of interest was the affect of different levels of these liquids in the feed mixture and the affect of different methods of adding these liquids to the mixer. Two experiments were run to determine these effects. The first experiment was a randomized complete block design with two fixed effects. Hemicellulose extract and cane molasses were mixed with wheat bran at levels of 10%, 15%, 20%, 25% and 30%. These 10 mixing tests (two treatments and five levels) were run in a random order. The tests were then repeated twice to give a total of three replications of each test. In all of these tests the liquids were added to the mixer in the same way.

A second experiment was carried out to determine the

affect of different methods of adding the liquid to the mixer. This experiment was of randomized complete block design with three fixed effects. Hemicellulose extract and cane molasses were used at the 10% and 25% levels. These levels were chosen after examining the results of the first experiment. In these tests two additional methods were used to add the liquids to the mixer. The three methods of addition are fully explained later when the procedure of the mixing tests is described.

In the first experiment, the moisture tracer method and the sieving method as well as the chloride tracer method of determining the liquid distribution in a feed mixture were used to make comparison of these three methods.

#### Preparation of Liquid Ingredients Used in Mixing Studies.

Before mixing experiments were begun, the liquids were prepared by adding the chloride tracer compounds; 7.5% potassium chloride by weight was added to the hemicellulose extract and 5.4% of a mixture of equal amounts of ammonium chloride and sodium chloride was added to the cane molasses. A sufficient amount of each liquid was prepared to complete both experiments. After the chloride compounds were completely dissolved, samples of the liquids were collected and analyzed to determine if the chlorides were uniformly distributed in the liquid. These analyses showed that the hemicellulose extract contained the desired 7.5% chloride expressed as potassium chloride and the

cane molasses contained the desired 7.5% chloride expressed as the mixture of ammonium chloride and sodium chloride. The viscosity of the hemicellulose extract used was 1400 centipoises at 30°C and the viscosity of cane molasses was 730 centipoises at 30°C. The surface tensions of hemicellulose extract and cane molasses were 57.0 and 55.6 dynes per centimeter, respectively. Viscosity and surface tension determinations on the prepared liquids showed that addition of the chloride compounds had not changed these physical properties. The liquids were stored in covered 50 gallon steel barrels. Each liquid was mixed thoroughly before being used in the mixing tests. During the period of time over which the mixing tests were being run, several samples of the liquids were analyzed to verify a uniform distribution of chloride in them.

Equipment and Procedures. The mixing tests were run in a small horizontal double ribbon batch mixer. The capacity of the mixer was approximately 5 cu. ft. when filled to a level one inch below the top of the outside ribbon. The dry ingredient used in the mixing tests was wheat bran. This material was used because it is a common ingredient in rations where cane molasses is used and it was readily available. Sixty pounds or about 5 cu. ft. of bran was used in each test. Table 2 shows the amounts of liquids that were mixed with the wheat bran to give the desired liquid levels in the total mixture. Although the total weight of the material in the mixer was not constant

Table 2. Amounts of Hemicellulose Extract and Cane Molasses Mixed with 60 Pounds of Wheat Bran.

Liquid Level in Per Cent of Total Weight in Mixer	Pounds of Liquid Added to Mixer
10	6.67
15	10.58
20	15.00
25	20.00
30	25.71

for all tests, the volume of the material being mixed was nearly constant for all tests since the addition of different amounts of liquid did not change the volume significantly.

The wheat bran was added to the mixer and allowed to mix for 15 seconds. This mixing caused the bran to become evenly distributed over the length of the mixer. With the mixer running the liquid was added at the top of the mixer. Figure 1 shows the equipment used to add the liquid. Air pressure was used to force the liquid from one to two 40 gallon pressure tanks into the mixer. One tank was used for hemicellulose extract, the other for cane molasses. The amount of liquid entering the mixer was controlled by the air pressure in the tank. This air pressure was adjusted by a pressure regulator. The time of liquid addition was 10 seconds for all tests. The liquid flow in 10 seconds from each tank at different pressures was determined. These values were plotted on graphs where the liquid flow was measured in pounds and the pressure was measured in pounds per square inch. For each test the pounds of liquid needed was known (Table 2). From the graphs, the pressure setting could be determined that would give the desired liquid flow in the 10 second period. The liquid flow was started by a fast acting ball valve in the liquid line just above the point of addition.

The liquids were added to the mixer by three different methods. In the first method the liquids were forced into the mixer at a point 15 inches from the discharge end from a single

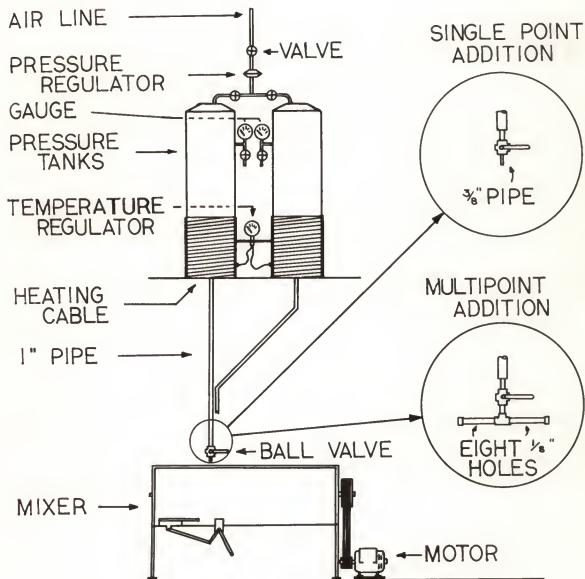


FIG. 1. EQUIPMENT USED FOR MIXING STUDIES.

3/8 inch opening. The liquids were added to the mixer without being heated. Their temperatures were between 22°C and 24°C. This method was referred to as single point addition.

The liquids were added in a second way by forcing them through eight 1/8 inch holes on a horizontal section of pipe. The length of this pipe was 12 inches and it was centered at the point of single point addition. The liquid was not heated. This method was referred to as multipoint addition.

The third method of addition was the same as the multipoint addition except that the liquids were heated. This heating was done by 80-foot 400 watt heating cables which were mounted to the lower section of the pressure tanks. The temperature of the liquids was controlled by a Mercoid Series "D" remote bulb temperature control. The liquids were heated to a temperature of 40°C. This method was referred to as multipoint heated liquid addition.

At the end of the 10 second addition period, the liquid flow was stopped and the material in the mixer was mixed for 45 seconds. Preliminary studies had indicated no difference in the mixing properties of hemicellulose extract and cane molasses when a uniform mixture with wheat bran was obtained. The mixing time in these preliminary studies had been 5 minutes. It seemed that a better comparison could be made if the mixing period was terminated before a uniform mixture was obtained. This 45 second mixing time was determined by an exploratory test using cane molasses at the 15% level. Samples were taken

after mixing periods of 15, 30, 60, and 90 seconds and analyzed for soluble chlorides. The coefficients of variation versus mixing time are plotted in Figure 2. The mixture began to approach uniformity after being mixed for 60 to 90 seconds. It was decided that a 45 second mixing time would give the best results.

At the end of the 45 second mixing period, sets of ten samples each were taken to be analyzed for moisture and soluble chlorides. The samples used for the moisture analysis were also used for the sieving test. The samples were taken from the surface of the material in the mixer with a small scoop which held approximately 20 grams. Two samples were taken side by side from the ten sampling points shown in Figure 3. The samples were taken from these points because the material in the mixer accumulated in the discharge end of the mixer as shown in Figure 4. The samples could not be taken with a probe because at the high liquid levels the material was very moist and would not fall into the probe. The samples were stored in sealed sample bottles until the analyses were run.

It is a common practice to weigh up a specified amount of each sample to be used for the analysis. Because there was a large amount of dust covered liquid balls present in some samples, splitting a sample might have introduced an additional sampling error. For this reason, the complete samples taken from the mixer with the scoop were analyzed.

The samples were analyzed for soluble chlorides according



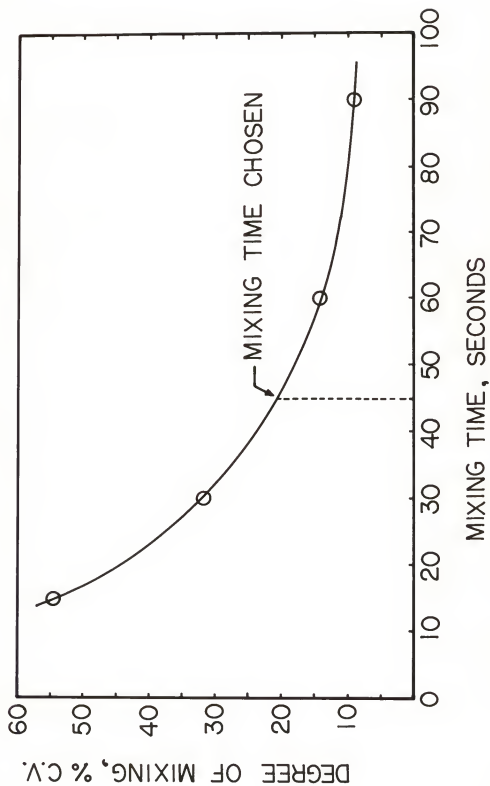


FIG. 2. EFFECT OF MIXING TIME ON DEGREE OF MIXING USING 15% CANE MOLASSES IN WHEAT BRAN.

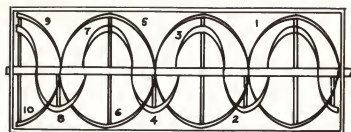


FIG. 3. TOP VIEW OF DOUBLE RIBBON HORIZONTAL MIXER INDICATING SAMPLING POSITIONS.

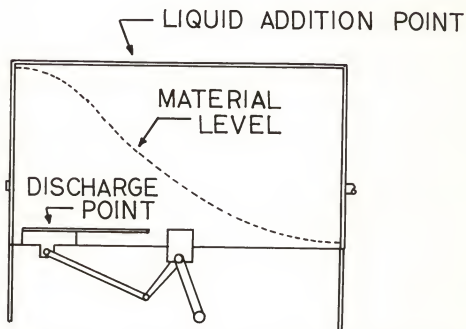


FIG. 4. SIDE VIEW OF MIXER INDICATING THE DISTRIBUTION OF THE MIXED MATERIAL.

to the method described by Luhman (4). However several aspects of his methods were changed so the method could be efficiently used in this study. The chloride analyses were run in the following manner. Samples were weighed on a torsion balance, and placed in 400 ml. beakers. Approximately 200 ml. of warm distilled water ( $40^{\circ}\text{C}$ ) was added to each beaker. Then 1 ml. of concentrated nitric acid was added. This mixture was stirred and allowed to stand for at least 10 minutes for complete solution of the chlorides. The sample was then titrated directly with a solution of silver nitrate. During the titration the sample was stirred with a magnetic stirrer. The potential between a calomel reference electrode and a silver electrode was read with a Beckman Zeromatic PH meter. A rapid decline in the millivolt reading indicated the titration end point. This end point had been previously determined for the different concentrations of silver nitrate that were used.

Different concentrations of silver nitrate were used because of the different chloride levels in the samples being analyzed. By using different concentrations of silver nitrate, approximately the same amount was used in each titration. Table 3 shows the theoretical level of chloride in the samples for each liquid level. These values include the 0.10% chloride present in the wheat bran. Table 3 also shows the normality of the silver nitrate solutions used. The amount of soluble chloride in the sample was determined by multiplying the amount of silver nitrate used by a conversion factor determined by the

Table 3. Theoretical Chloride Contents, Concentration of Silver Nitrate Solutions and Conversion Factors Used in Mixing Tests.

Liquid Level	Theoretical Chloride Content	Normality of $\text{AgNO}_3$	Conversion Factor
Hemicellulose Extract			
10	0.850 <sup>1</sup>	0.10	0.00745
15	1.225 <sup>1</sup>	0.15	0.01120
20	1.600 <sup>1</sup>	0.20	0.01490
25	1.975 <sup>1</sup>	0.25	0.01860
30	2.350 <sup>1</sup>	0.30	0.02240
Cane Molasses			
10	0.850 <sup>2</sup>	0.10	0.00559
15	1.225 <sup>2</sup>	0.15	0.00838
20	1.600 <sup>2</sup>	0.20	0.01190
25	1.975 <sup>2</sup>	0.25	0.01400
30	2.350 <sup>2</sup>	0.30	0.01680

<sup>1</sup>Expressed as % potassium chloride.

<sup>2</sup>Expressed as % of a mixture of equal amounts of ammonium chloride and sodium chloride.

principles of quantitative analysis. Table 3 also shows these conversion factors. The amount of soluble chlorides was converted to a percentage value by dividing it by the weight of the sample. The per cent of soluble chlorides was expressed as per cent potassium chloride for tests using hemicellulose extract and as per cent of the ammonium chloride-sodium chloride mixture for tests using cane molasses. The ten percentage values of chloride in the samples from each mixing test were used to calculate the coefficient of variation for each test.

The second set of samples was placed in large aluminum moisture dishes of known weight. The samples were then weighed and dried in a forced air oven for two hours at 135°C. The samples were cooled for about two hours in a desiccator and reweighed. The moisture content in per cent was determined by dividing the weight lost during the drying by the weight of the original sample. These values were used to calculate the coefficient of variation for each of the mixing tests.

The ten dried samples from each mixing test were combined to form a single sample that was used in the sieving test. The drying of the samples hardened the dust covered liquid balls. These balls were then separated by sieving the composite sample on a Ro-tap sieve shaker for five minutes. Preliminary studies had been run using a Tyler number 6 sieve with 0.131 inch openings. This size of opening had been satisfactory for mixtures of wheat bran and low liquid levels. However, during the first tests of the experiment it was

noticed that the number 6 sieve was not giving accurate results. This difficulty arose from the fact that after the drying process the bran particles were being held together by the dried liquid when high amounts of liquid were present. These particles would not pass through the openings of the number 6 sieve. The sieve was separating more than the large liquid balls. It was found that a Tyler number  $3\frac{1}{2}$  sieve with 0.221 inch opening did a much better job of separating only the large dust covered liquid balls. Plate 1 shows some of the dust covered liquid balls separated by the number  $3\frac{1}{2}$  sieve. Unfortunately this was determined after the first two replications were run, but the number  $3\frac{1}{2}$  sieve was used for the third replication of the mixing test.

#### PELLETING STUDIES

The purpose of these experiments was to compare the production of feed pellets containing liquid hemicellulose extract from Mississippi and California, dried hemicellulose extract, and cane molasses. Of primary concern were the energy required to produce the pellets and the pellet durability. Tests were also conducted to compare different levels of dried hemicellulose extract in the formula and to compare the binding characteristics of dried hemicellulose extract with that of a common lignin binder.

Equipment and Procedures. The pelleting in all of the experiments was done with a 25 horsepower California Master

## PLATE 1

Fig. a



Fig. b



model pellet mill. The pellets were cooled in a California vertical pellet cooler, size 2-B. The pelleting energy required was measured with a recording watt-hour meter. The pellet mill was started with the pellet cooler running on continuous discharge to keep the cooler empty during the period it took to obtain a stable pelleting rate and temperature. When stable operating conditions were obtained the pellet cooler was set on automatic discharge and the recording watt-hour meter was started. The pellets obtained during the start-up period were sacked off. With the cooler on the automatic discharge the hot pellets are collected in the cooling chamber. For all of the experiments, 1000 pound batches were pelleted. This was approximately enough pellets to fill the cooling chamber. If the chamber became full, the cooled pellets were automatically discharged from the cooler to permit more pellets to enter the cooling chamber. When the pelleting was completed the pellets were allowed to cool for a period of time in the chamber. They were then discharged from the cooler and passed over a 4-mesh scalping screen before dropping into the sacking bin. The whole pellets were sacked off and weighed. The broken pellets passing through the scalping screen were collected and later pelleted again. At the end of each batch the watt-hour meter reading was recorded. The energy requirements in watt-hours per ton was determined by dividing the watt-hours used by the amount of whole pellets produced. Three samples of cooled pellets were collected after each test to determine the



pellet durability index.

Effect of High Levels of Hemicellulose Extract. The formula used in this experiment was a high alfalfa ration for wintering beef cattle (formula AH-92). Hemicellulose extract was used as an energy source. Each test ration had approximately the same energy content. The rations contained 13.6% dried hemicellulose extract from Mississippi and California, 20% liquid hemicellulose extract from Mississippi and California, and 20% cane molasses. A ration containing 13.6% dried hemicellulose extract and 3% water was also used. The formulation of the rations used in this experiment are given in Table 4.

The dry ingredients were mixed in a 500 pound horizontal batch mixer and transferred to the continuous mixing system where the liquid hemicellulose extract or cane molasses was added in a horizontal double paddle mixer. The feed was then transferred to the pellet mill holding bin. In the rations with no liquid hemicellulose extract and with dried hemicellulose extract, the material was transferred directly from the batch mixer to the pellet mill holding bin. In the ration containing the liquid ingredients, the material was transferred into the holding bin above the pellet mill at the same rate that it was pelleted to eliminate the possibility of the feed becoming lodged in the bin. Also a special screw feeder had to be used to feed the material from the pellet mill conditioning chamber to the pellet die. A 3/4 in. by 3½ in.

Table 4. Formulation of High Alfalfa Cattle Cubes (AH-92)

Ingredients	Per Cent of Ration by Weight							
	AH-92-A : AH-92-B : AH-92-C : AH-92-D : AH-92-E : AH-92-F							
Soybean Oil Meal	35	37	37	38	38	38	38	38
Dehydrated Alfalfa Meal	23	23	23	23	23	23	23	23
Ground Sorghum Grain	32	16.4	16.4	9	9	9	9	9
Urea	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Dicalcium Phosphate	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
Salt	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Vitamin-Mineral Premix	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Dried Hemicellulose Extract (Miss.)	13.6							
Dried Hemicellulose Extract (Cal.)			13.6					
Hemicellulose Extract (Miss.)				20.0				
Hemicellulose Extract (Cal.)					20.0			
Cane Molasses	100	100	100	100	100	100	100	100
								20.0
								100

die was used. The temperature of the mash leaving the conditioning chamber was about 50°C. in all tests except in the test using the ration with no hemicellulose extract or cane molasses the temperature was 70°C. The pellets were cooled for 20 minutes before they were discharged from the cooler. Each test was replicated at least three times.

Effect of Low Levels of Hemicellulose Extract. The basic formula used in this experiment was a swine lactation ration containing 4% added fat. To this formula 1%, 2%, and 3% levels of dried hemicellulose extract, 3% levels of liquid hemicellulose extract from Mississippi and California, and 3% cane molasses was added. The formulation of these rations are given in Table 5.

All of the rations were mixed in a 500 pound horizontal batch mixer and transferred to the pellet mill. A 3/16 in. by 2 in. pellet die was used. The pellets were cooled for a period of 10 minutes. Three replications of each test were run except for the tests using the rations containing 1% and 3% dried hemicellulose extract. Only one test was run on each of these rations.

Comparison of Dried Hemicellulose Extract and a Lignin Binder. The basic formula used in this experiment was a swine finishing ration containing a high level of ground sorghum grain. Tests were run with 2% dried hemicellulose extract, 2%



lignin binder (calcium lignosulphonate), and 1% of both dried hemicellulose extract and lignin binder added to the basic formula. The formulation for the rations used in this experiment are shown in Table 6. The pellet die and the procedure used was the same as in the previous experiment. Each test was replicated two times.

Table 6. Formulation of Swine Finishing Ration (S-80)

Ingredients	Per Cent of Ration by Weight			
	S-80-A	S-80-B	S-80-C	S-80-C
Ground Sorghum Grain	89.8	87.8	87.8	87.8
Soybean Oil Meal	7.0	7.0	7.0	7.0
Dicalcium Phosphate	1.0	1.0	1.0	1.0
Ground Limestone	0.7	0.7	0.7	0.7
Salt	0.5	0.5	0.5	0.5
Vitamin-Mineral Premix	1.0	1.0	1.0	1.0
Dried Hemicellulose Extract		2.0		1.0
Lignin Binder			2.0	1.0
	100	100	100	100

## RESULTS AND DISCUSSION

### MIXING STUDIES

The results of the first mixing experiment are shown in Table 7. The values shown are coefficients of variation for the chloride tracer and moisture tracer methods. The values for the sieve analysis are the percentages retained over a Tyler number 3½ sieve. Table 8 shows the results of an analysis of variance that was performed on the coefficients of variation obtained using the chloride tracer method for determining degree of mixing. The analysis treated two variables; liquid treatment and liquid level--with one two way interaction. This permitted the statistical testing of mixing uniformity against liquid treatments and liquid levels.

The results of the statistical analyses indicated no significant differences among the two liquid treatments and among the five liquid levels. There was, however, a significant interaction (0.05 level) between liquid treatments and liquid levels. A t-test was performed to determine any difference among liquid levels separated by liquid treatments. This test showed no significant difference (0.05 level) either among different levels of hemicellulose extract or among different levels of cane molasses.

Linear correlation analyses for correlating laboratory methods, using coefficient of variation as the criterion, yielded correlation coefficients of 0.93 and 0.52 for moisture

Table 7. Results of Chloride Tracer Method, Moisture Tracer Method and Sieve Analysis of Five Levels of Hemicellulose Extract and Cane Molasses Mixed with Wheat Bran.

Liquid Level	Mixing Replication	Laboratory Methods		Sieve Analysis % over No. 3½
		Chloride Tracer, %C.V.	Moisture Tracer, %C.V.	
Hemicellulose Extract				
10	1	36.7	7.39	
	2	22.9	5.04	
	3	19.9	5.52	7.61
15	1	18.8	7.64	
	2	24.5	7.77	
	3	20.7	5.61	8.36
20	1	26.7	5.90	
	2	15.3	4.20	
	3	17.7	5.25	10.10
25	1	11.8	3.91	
	2	15.1	5.17	
	3	23.1	7.74	11.74
30	1	11.5	5.55	
	2	11.8	5.54	
	3	18.1	8.09	8.41
Cane Molasses				
10	1	12.1	2.57	
	2	14.1	1.51	
	3	19.7	3.11	1.78
15	1	14.8	3.23	
	2	23.8	4.68	
	3	12.3	2.57	2.51
20	1	23.1	6.28	
	2	28.9	7.74	
	3	14.5	3.53	4.95



Table 7 (continued)

Liquid Level	Mixing Replication	Laboratory Methods		
		Chloride Tracer. %C.V.	Moisture Tracer. %C.V.	Sieve A % over
25	1	41.1	11.00	
	2	36.7	10.60	
	3	14.8	6.24	5.
30	1	22.5	8.32	
	2	42.0	12.47	
	3	9.5	2.95	5.

Table 8. Tabulation of Analysis of Variance Results Testing Degree of Mixing Determined by the Chloride Tracer Method with Respect to Liquid Treatments and Liquid Levels.

	Sums of Squares	Degrees of Freedom	Mean Square	F-Test
Total	2274.50	29		
Treatment	41.53	1	41.53	0.611
Level	84.24	4	21.06	0.310
Treatment by level	788.30	4	197.07	2.897 <sup>1</sup>
Error	1360.43	20	68.02	

<sup>1</sup>Significantly different at the 0.05 level.

tracer method versus chloride tracer method for cane molasses and hemicellulose extract, Figures 5 and 6 respectively. The sieve analysis which was run only on the last replication of the tests, showed that there was a larger per cent of liquid balls present in the tests with hemicellulose extract than in the tests using cane molasses. This fact may have caused greater sampling errors and moisture assay errors which resulted in a poor correlation of the moisture tracer and chloride tracer methods in the tests with hemicellulose extract.

The results of the second mixing experiment comparing three methods of adding 10% and 25% of either hemicellulose extract or cane molasses to the mixer are shown in Table 9. The values shown are the coefficients of variation obtained using the chloride tracer laboratory method. An analysis of variance was performed in these results treating three variables; liquid treatments, liquid levels, and method of liquid addition--with three two way interactions and one three way interaction. The results of this statistical analysis are shown in Table 10.

The analysis of variance showed no significant difference among the two liquid treatments. However, a significant difference (0.05 level) was noted among the two liquid levels. The two liquid levels used in this experiment were chosen after the results of the first experiment were obtained. Even though, in the first experiment, the affect of level was not significant the 10% and 25% levels seemed to give the greatest difference

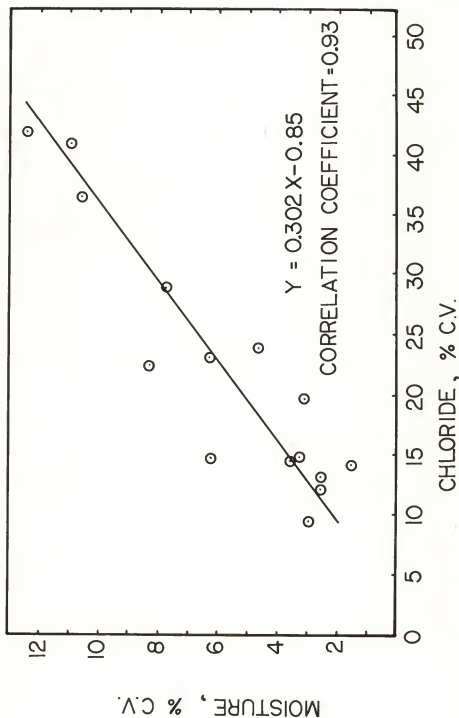


FIG. 5. LINEAR REGRESSION ANALYSIS OF MOISTURE TRACER  
VERSES CHLORIDE TRACER FOR MEASURING DEGREE  
OF MIXING OF CANE MOLASSES.

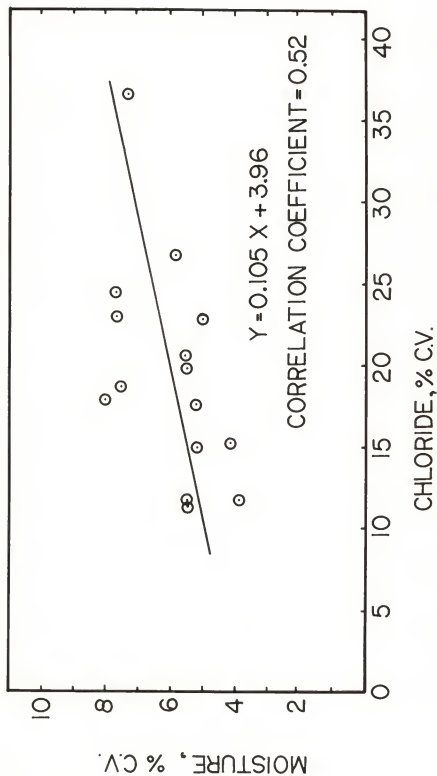


FIG. 6. LINEAR REGRESSION ANALYSIS OF MOISTURE TRACER VERSUS CHLORIDE TRACER FOR MEASURING DEGREE OF MIXING OF HEMICELLULOSE EXTRACT.

Table 9. Results of Chloride Tracer Method for Measuring Degree of Mixing of Two Levels of Hemicellulose Extract and Cane Molasses with Wheat Bran Using Three Methods of Liquid Addition

Liquid Level	Mixing Replication	Methods of Liquid Addition		
		Single Point Addition	Multipoint Addition	Multipoint Heated Liquid Addition
Hemicellulose Extract				
10	1	36.7	18.5	32.6
	2	22.9	22.6	23.6
	3	19.9	27.9	34.8
25	1	11.8	13.5	11.9
	2	15.1	10.8	19.4
	3	23.1	6.9	14.5
Cane Molasses				
10	1	12.1	11.3	14.7
	2	14.1	14.1	25.6
	3	19.1	13.2	41.1
25	1	41.1	8.3	9.7
	2	36.7	8.2	18.5
	3	14.8	9.5	17.3

Table 10. Tabulation of Analysis of Variance Results Testing Degree of Mixing Determined by the Chloride Tracer Method with Respect to Liquid Treatment, Liquid Level, and Method of Liquid Addition.

	Sums of Squares	Degrees of Freedom	Mean Square	F-Test
Total	3241.59	35		
Treatment	37.01	1	37.01	0.725
Level	362.90	1	362.90	7.112 <sup>1</sup>
Method of Addition	568.05	2	284.03	5.566 <sup>1</sup>
Treatment by Level	340.40	1	340.40	6.671 <sup>1</sup>
Treatment by Method of Addition	83.53	2	41.77	0.819
Level by Method of Addition	421.54	2	210.77	4.130 <sup>1</sup>
Treatment by Level by Method of Addition	203.54	2	101.77	1.994
Error	1224.62	24	51.03	

<sup>1</sup>Significantly different at the 0.05 level.

between the uniformity of the mixtures containing the two liquids. So these levels were used in the second experiment. Therefore, because of the way in which these two levels were chosen and possibly because of the additional methods of addition, there is a significant difference among levels in the second experiment. There was also a significant difference among the methods of liquid addition. Significant interactions were obtained between liquid treatments and liquid levels, and between liquid levels and methods of addition.

#### PELLETING STUDIES

Effect of High Levels of Hemicellulose Extract. The results of this experiment are shown in Table 11. Each value for pellet durability index and kilowatt-hours per ton is the average of at least three pelleting tests of 1000 pounds each.

During the pelleting process, a little difficulty was encountered in maintaining a constant load on the pellet mill. The load was controlled by adjusting the feed rate and steam addition into the conditioning chamber so that a constant ammeter reading of 30 amperes is obtained. The trouble in this experiment was encountered only in the tests where the ration with a 20% liquid level was being pelleted. Poor blending of the liquid with the dry ingredients may have been a cause of this trouble. However, by continual adjustment of the feed rate and the steam rate, the ammeter reading was held fairly constant during the tests. This difficulty could not be



Table 11. Effect of High Levels of Hemicellulose Extract

Treatment	Replications	Formula	PDI <sup>1</sup>	KWH/TON <sup>2</sup>
None	4	AH-92-A	9.57	9.31
13.6% Dried Hemicellulose Extract (Miss.)	4	AH-92-B	9.77	15.18
13.6% Dried Hemicellulose Extract (Cal.)	3	AH-92-C	9.79	9.30
20% Hemicellulose Extract (Miss.)	3	AH-92-D	9.81	7.70
20% Hemicellulose Extract (Cal.)	3	AH-92-E	9.81	6.13
20% Cane Molasses	3	AH-92-F	9.84	6.05
13.6% Dried Hemicellulose Extract (Miss.) and 3% Water	4	AH-92-B	9.82	9.09

<sup>1</sup>Pellet durability index

<sup>2</sup>Kilowatt-hours per ton

attributed to any one of the three types of liquids used in the experiment.

The data shown in Table 11 indicates that the addition of dried hemicellulose extract, liquid hemicellulose extract, and cane molasses did improve the pellet durability index. A t-test (0.01 level) showed no differences in the durability of pellets containing these ingredients. Dried hemicellulose extract from Mississippi caused an increase in the power required, while the power required to pellet the ration containing dried hemicellulose from California was the same as that required by the formula with no hemicellulose extract. The formulas containing the 20% liquid levels required less power than the formulas with no hemicellulose extract. A t-test (0.01 level) showed no significant difference in the power required by the rations with the different liquid ingredients. A pelleting test was run with 3% water added to the formula with 13.6% dried hemicellulose extract from Mississippi to determine if the power required could be reduced in this manner. The addition of water did reduce the power required. This high power required when pelleting the ration containing 13.6% dried hemicellulose extract from Mississippi is of little concern because dried hemicellulose extract is very seldom used at such a high level in a ration. It is primarily used at low levels as a pellet binder. Low levels of hemicellulose extract were evaluated in the following experiment.

Effect of Low Levels of Hemicellulose Extract. The results of this experiment are given in Table 12. There was an improvement in the pellet durability index as the level of dried hemicellulose extract was increased from zero to 3%. The 3% level of liquid in the formula gave an increased pellet durability index, however, a t-test (0.01 level) showed no significant difference in the durability of pellets containing the three different liquid ingredients. There was no significant difference (0.01 level) in the power required by any of the rations pelleted in this experiment.

Comparison of Dried Hemicellulose Extract and a Lignin Binder. Table 13 shows the results of this experiment. Pellets with 2% levels of dried hemicellulose extract and lignin binder and a 1% level of both dried hemicellulose extract and lignin binder had improved pellet durabilities over the pellets with no dried hemicellulose extract or lignin binder. A t-test (0.01) showed no significant difference in the durability of pellets containing 2% of either dried hemicellulose extract or lignin binder. There was no significant difference (0.01 level) in the power required to pellet any of the rations used in this experiment.

## ACKNOWLEDGMENTS

The author wishes to express his sincere appreciation to Dr. Harry B. Pfost for his guidance during the research and preparation of this manuscript.

Appreciation is also expressed to Dr. William Hoover, Head of the Department of Grain Science and Industry, to Dr. John A. Shellenberger, former Head of this Department, and to the entire staff of this department for their guidance and for providing the equipment and materials needed to carry on this research.

Acknowledgment is also made to Masonite Corporation for their interest and support in making this research possible.

PROCESSING FEEDS CONTAINING HEMICELLULOSE EXTRACT

by

Montford Lee Carpenter

B.S., Kansas State University, 1965

---

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

Kansas State University

Manhattan, Kansas

1968

Hemicellulose extract is a by-product of an industrial process for the production of hardboard. It is produced in both liquid and dried form in Laurel, Mississippi and in liquid form in Ukiah, California. Liquid hemicellulose extract is used as an energy source much the same as cane molasses in livestock feeds. The liquid product from Mississippi has a higher viscosity than the California product whose viscosity is similar to cane molasses. Dried hemicellulose extract is used primarily as a binder in the production of feed pellets. Because of its recent introduction as a feed ingredient it was desirable to evaluate the affect of hemicellulose extract on the mixing and pelleting of feeds.

Before any mixing tests were made a method was developed to measure the distribution of liquid hemicellulose extract and cane molasses in a feed mixture. It was found that certain chloride compounds had no affect on the viscosity or surface tension of hemicellulose extract or cane molasses when dissolved in these liquids. It was assumed that these compounds would have no affect on the mixing properties of the liquids and could be used as a tracer for determining the distribution of the liquids in a feed mixture.

This chloride tracer method showed that there was no difference in the mixing properties of liquid hemicellulose extract and cane molasses when mixed with wheat bran at 10%, 15%, 20%, 25% and 30% levels. Using moisture as a tracer gave results similar to the chloride tracer method for cane molasses.

However, the results differed in the case of hemicellulose extract. A sieving test to measure the amount of large dust covered liquid balls in the feed mixture showed that the hemicellulose extract-wheat bran mixture gave more of these balls but it did not give results that could be used as an evaluation of the distribution of the liquids in the feed mixture.

The results of a second mixing experiment again showed no difference in the mixture obtained when either hemicellulose extract or cane molasses was added to the mixer by three different methods. Adding the liquid from several points along the top of a horizontal mixer gave better mixtures than adding the liquid from a single point or even adding the liquid from several points after it was heated.

Pelleting studies showed that pellets from rations containing high levels (20%) of either liquid hemicellulose extract or cane molasses had similar durability indexes and required similar amounts of power to produce. The same results were obtained for rations with low levels (3%) of either liquid hemicellulose extract or cane molasses. It was found that the pellet durability index increased as the level of dried hemicellulose extract in the ration increased from 0 to 3% without an increase in the power requirements. Dried hemicellulose extract and a lignin binder gave similar pellet durabilities and power requirements when used at 2% levels in the ration.